



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of  
Hiroyasu Nishida, et al.

Group Art Unit: 1712  
Examiner: Daniel S. Metzmaier

Serial No. 09/038,230

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For: Inorganic Compound Sol Modified by Organic Compound

Honorable Commissioner of Patents and Trademarks  
United States Patent and Trademark Office  
Washington, D. C. 20231

Sir:

DECLARATION UNDER 37 CFR 1.132

I, Hiroyasu Nishida, hereby declare as follows:

1. I am a citizen of Japan, and residing at 13-2, Kitaminato-machi, Wakamatsu-ku, Kitakyushu-shi, Fukuoka, Japan.

In March 1969, I was graduated from Tokyo University of Science, and received a degree of Bachelor of Science.

In March 1971, I was graduated from the graduate course of Tokyo University of Science, and received a degree of Master of Science from Tokyo University of Science.

Since April 1971, I have been an employee of Catalyst & Chemicals Industries Co., Ltd. Simultaneously, I had been assigned to Wakamatsu Plant and engaged in the development of novel inorganic materials, especially the production of the fine inorganic powders.

2. I am a co-inventor of the invention described in the specification of the above-identified application.
3. The following Experimental Test was carried out in order to demonstrate the unobviousness of the present invention.

## **Experiments**

### Experiment 1

500 g of a silica/alumina water dispersion sol (Catalysts & Chemicals Industries Co., Ltd., Cataloid-SN, weight ratio of  $\text{SiO}_2/\text{Al}_2\text{O}_3=285.7$ , average particle size: 12 nm and solid content: 20% by weight) was used as a starting material and subjected to a solvent displacement by means of an ultrafilter to thereby replace the water by methanol. Thus, there was obtained a methanol dispersion sol having a solid content of 30% by weight.

300 g of this sol was mixed with 2700 g of ethanol, and 8.4 g of tetraethoxysilane (molecular polarizability of  $11.9 \times 10^{-30} \text{ C}^2\text{m}^2\text{J}^{-1}$ ) was added to the mixture.

The mixture was agitated for 1 hr, thereby obtaining a sol containing silica/alumina particulates whose surface was modified by tetraethoxysilane.

196 g of ethylene glycol was added to the obtained sol and heated at 60 °C. for 1 hr. Thereafter, ethanol was removed in vacuum by the use of a rotary evaporator, thereby obtaining a surface-modified silica/alumina sol of 30% by weight in solid content which contained ethylene glycol (dielectric constant of 38) as a dispersion medium.

The sol stability I and II of the obtained surface-modified silica/alumina sol was evaluated in the same manner as in Example 1 of the present specification.

The viscosity of the obtained sol in the sol stabilities I and II was improved at 15days, the sol was gelated at 19 days.

## Experiment 2

A surface-modified silica/alumina sol was obtained in the same manner as in Experiment 1, except that 8.4 g of 3-(vinylbenzylaminopropyl) trimethoxysilane (molecular polarizability of  $8.31 \times 10^{-35} \text{ C}^2\text{m}^2\text{J}^{-1}$ ) was used instead of tetraethoxysilane.

The sol stability I and II of the obtained surface-modified silica/alumina sol was evaluated in the same manner as in above Experiment 1.

The viscosity of the obtained sol in the sol stabilities I and II was improved at 20 days, the sol was gelated at 31 days.

## Experiment 3

A surface-modified silica/alumina sol was obtained in the same manner as in Experiment 1, except that 3.0 g of sodium dodecylbenzenesulfonate (surface active agent) was used instead of tetraethoxysilane.

The sol stability I and II of the obtained surface-modified silica/alumina sol was evaluated in the same manner as in above Experiment 1.

The viscosity of the obtained sol in the sol stabilities I and II was improved at 1 days, the sol was gelated at 3 days.

In any Experiments, the viscosity of the soles was improved and the soles were gelated soon. As a result the stable sol cannot be obtained.

4. From the result of the above Experiment and based on my best knowledge and experience on the inorganic material development, I conclude as follows.

It is clear that by the common modifiers, particulates are not always excellent in stability in sol or solution in the presence of acids, alkalis or surface active agent exist.

The undersigned declares further that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

This 12 day of November 2004

Hiroyasu Nishida

Hiroyasu NISHIDA